

FORM PTO-1390 U.S. DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE (REV. 10-95)		ATTORNEY'S DOCKET NUMBER
TRANSMITTAL LETTER TO THE UNITED STATES DESIGNATED/ELECTED OFFICE (DO/EO/US) CONCERNING A FILING UNDER 35 U.S.C. § 371		J6547(C)
		U.S. APPLICATION NO. (If known, see 37 CFR § 1.5) 10/089648
INTERNATIONAL APPLICATION NO.	INTERNATIONAL FILING DATE	PRIORITY DATE CLAIMED
PCT/EP00/09144	18 SEPTEMBER 2000	1 OCTOBER 1999
TITLE OF INVENTION		
ANTIPERSPIRANT COMPOSITIONS COMPRISING MICROEMULSIONS		
APPLICANT(S) FOR DO/EO/US		
MA, ZHUNING ET AL.		
Applicant herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information:		
<ol style="list-style-type: none">1. <input checked="" type="checkbox"/> This is a FIRST submission of items concerning a filing under 35 U.S.C. § 371.2. <input type="checkbox"/> This is a SECOND or SUBSEQUENT submission of items concerning a filing under 35 U.S.C. § 371.3. <input type="checkbox"/> This express request to begin national examination procedures (35 U.S.C. §371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. §371(b) and PCT Articles 22 and 39(I).4. <input checked="" type="checkbox"/> A proper DEMAND for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.5. <input checked="" type="checkbox"/> A copy of the International Application as filed (35 U.S.C. §371(c)(2))<ol style="list-style-type: none">a. <input type="checkbox"/> is transmitted herewith (required only if not transmitted by the International Bureau).b. <input checked="" type="checkbox"/> has been transmitted by the International Bureau.c. <input type="checkbox"/> is not required, as the application was filed in the United States Receiving Office (RO/US).6. <input type="checkbox"/> A translation of the International Application into English (35 U.S.C. §371(c)(2)).7. <input checked="" type="checkbox"/> Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. §371(c)(3))<ol style="list-style-type: none">a. <input type="checkbox"/> are transmitted herewith (required only if not transmitted by the International Bureau).b. <input type="checkbox"/> have been transmitted by the International Bureau.c. <input type="checkbox"/> have not been made, however, the time limit for making such amendments has NOT expired.d. <input checked="" type="checkbox"/> have not been made and will not be made.8. <input type="checkbox"/> A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. §371(c)(3)).9. <input checked="" type="checkbox"/> An oath or declaration of the inventor(s) (35 U.S.C. §371(c)(4)).10. <input type="checkbox"/> A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. §371(c)(5)).		
Items 11. To 16. Below concern document(s) or information included:		
<ol style="list-style-type: none">11. <input type="checkbox"/> An Information Disclosure Statement under 37 C.F.R. §§ 1.97 and 1.98.12. <input type="checkbox"/> An assignment document for recording. A separate cover sheet in compliance with 37 C.F.R. §§3.28 and 3.31 is included.13. <input checked="" type="checkbox"/> A FIRST preliminary amendment. <input type="checkbox"/> A SECOND or SUBSEQUENT preliminary amendment.14. <input type="checkbox"/> A substitute specification.15. <input type="checkbox"/> A change of power of attorney and/or address letter.16. <input type="checkbox"/> Other items or information:		

U.S. APPLICATION NO. (if known) 10/089648	INTERNATIONAL APPLICATION NO. PCT/EP00/09144	ATTORNEY'S DOCKET NUMBER J6547(C)
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17. <input checked="" type="checkbox"/> The following fees are submitted:				CALCULATIONS PTO USE ONLY	
BASIC NATIONAL FEE (37 CFR §1.492(a)(1)-(5)): Search Report has been prepared by the EPO or JPO \$ International preliminary examination fee paid to USPTO \$ (37 CFR §1.482) No international preliminary examination fee paid to USPTO \$ (37 CFR §1.482) but international search fee paid to USPTO (37 CFR §1.445(a)(2)) Neither international preliminary examination fee (37 CFR §1.482) \$ nor international search fee (37 CFR §1.445(a)(2)) paid to USPTO International preliminary examination fee paid to USPTO \$ (37 CFR §1.482) and all Claims satisfied provisions of PCT article 33(2)-(4).					
ENTER APPROPRIATE BASIC FEE AMOUNT =				\$890.00	
Surcharge of \$130.00 for furnishing the oath or declaration later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 CFR §1.492(e)).					
CLAIMS	NUMBER FILED	NUMBER EXTRA	RATE		
Total Claims	16 - 20 =		X \$18.00		
Independent Claims	1 - 3 =		X \$80.00		
MULTIPLE DEPENDENT CLAIM(S) (if applicable)			X \$270.00		
TOTAL OF ABOVE CALCULATIONS =				\$890.00	
Reduction of 1/2 for filing by small entity, if applicable. A Verified Small Entity Statement must also be filed (Note 37 C.F.R. §§ 1.9, 1.27, 1.28).					
SUBTOTAL =					
Processing fee of \$130.00 for furnishing the English translation later than <input type="checkbox"/> 20 <input type="checkbox"/> 30 months from the earliest claimed priority date (37 C.F.R. § 1.492(f)).					
TOTAL NATIONAL FEE =					
Fee for recording the enclosed assignment (37 C.F.R. § 1.21(h)). The assignment must be accompanied by an appropriate cover sheet ((37 C.F.R. §§ 3.28, 3.31). \$40.00 per property.					
TOTAL FEES ENCLOSED =				\$890.00	
				Amount to be refunded:	
				Charged:	

- a. ☐ A check in the amount of _____ to cover the above fees is enclosed.
- b. ☒ Please charge Deposit Account No. 12-1155 in the amount of \$890.00 to cover the above fees. Triplicate copies of this letter are enclosed.
- c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 12-1155. Triplicate copies of this letter are enclosed.

Customer Number:



00201

PATENT TRADEMARK OFFICE

NOTE: Where an appropriate time limit under 37 C.F.R. §§ 1.494 or 1.495 has not been met, a petition to revive (37 C.F.R. §§ 1.137(a) or (b)) must be filed and granted to restore the application to pending status.

Respectfully submitted,

Rimma Mitelman
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SCANNED, #12

10089648-072902

10/089648

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PATENT
#99-0080-HC
Case #J6547(C)

Express Mail Label No.: ET 506 459 646 US

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant: Ma et al.
Deposited: April 1, 2002
For: Antiperspirant Compositions Comprising Microemulsions

Edgewater, New Jersey 07020
April 1, 2002

PRELIMINARY AMENDMENT

Assistant Commissioner for Patents
Washington, D.C. 20231

Sir:

With regard to the above-identified application filed concurrently herewith, please amend the following:

In the Claims:

Please enter the following amended claims:

3. (Amended) A composition in accordance with claim 1 characterised in that said antiperspirant salt is a zirconium salt complexed with aluminum salts having coordinated or bound water.
4. (Amended) A composition in accordance with claim 1 characterised in that said antiperspirant salt is present in the aqueous phase at from about 1 to about 60%.

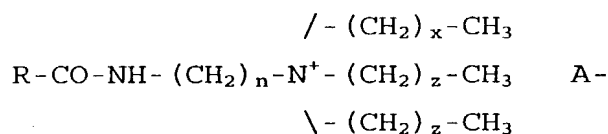
6. (Amended) A composition in accordance with claim 1 characterised in that said aqueous phase further comprises a buffer, a glycol, a sugar, a cyclodextrin, a preservative, an antimicrobial, a chelating agent, a water-soluble polymer, an anticholinergic, a monovalent salt, a divalent salt, a trivalent salt, fragrances or mixtures thereof.

7. (Amended) A composition in accordance with claim 1 cit said aqueous phase is present at about 1% to about 60%, more preferably at 5% to 30%, and most preferably at 10 to 25%.

8. (Amended) A composition in accordance with claim 1 characterised in that said cosmetic oil comprises esters, ethers, long chain alcohols, or ethoxylated alcohols, hydrocarbons, fatty acids, monoglycerides, diglycerides triglycerides, fragrances and volatile or non-volatile silicone fluids, and cholesterol.

10. (Amended) A composition in accordance with claim 8 characterised in that said non-volatile silicone is phenyl tris(trimethylsiloxy)silane.

13. (Amended) A composition in accordance with claim 1 characterised in that the cationic quaternary ammonium surfactant has the following structure:



wherein n is one to six.

x is zero to three

y is zero to three

z is zero to three

with the proviso that $x+y+z \leq 6$

REMARKS

The present amendment is submitted to eliminate multiple dependencies and to correct minor typographical errors. The amendments were not intended to and should not be construed to have been made for any reasons related to patentability of the claims.

Attached hereto is a marked-up version of the changes made to the claims by the current amendment. The attachment is captioned "Version with Markings to Show Changes Made".

Respectfully submitted,



Rimma Mitelman
Registration No. 34,396
Attorney for Applicant(s)

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VERSION WITH MARKINGS TO SHOW CHANGES MADE

3. (Amended) A composition in accordance with claim 1 ~~or 2~~ characterised in that said antiperspirant salt is a zirconium salt complexed with aluminum salts having coordinated or bound water.

4. (Amended) A composition in accordance with ~~any preceding claim~~ claim 1 characterised in that said antiperspirant salt is present in the aqueous phase at from about 1 to about 60%.

6. (Amended) A composition in accordance with ~~any preceding claim~~ claim 1 characterised in that said aqueous phase further comprises a buffer, a glycol, a sugar, a cyclodextrin, a preservative, an antimicrobial, a chelating agent, a water-soluble polymer, an anticholinergic, a monovalent salt, a divalent salt, a trivalent salt, fragrances or mixtures thereof.

7. (Amended) A composition in accordance with ~~any preceding claim~~ claim 1 cit said aqueous phase is present at about 1% to about 60%, more preferably at 5% to 30%, and most preferably at 10 to 25%.

8. (Amended) A composition in accordance with ~~any preceding claim~~ claim 1 characterised in that said cosmetic oil comprises esters, ethers, long chain alcohols, or ethoxylated alcohols, hydrocarbons, fatty acids, monoglycerides, diglycerides triglycerides, fragrances and volatile or non-volatile silicone fluids, and cholesterol.

10. (Amended) A composition in accordance with claim 8 ~~or 9~~ characterised in that said non-volatile silicone is phenyl tris(trimethylsiloxy)silane.

ANTIPERSPIRANT COMPOSITIONS COMPRISING MICROEMULSIONS5 Field of the Invention

This invention is related to microemulsions that contain cosmetically active ingredients. In a preferred embodiment, this invention is related to antiperspirant
10 salt-containing microemulsions that are stable, clear liquids and are easy and inexpensive to produce.

Background of the Invention

15

The microemulsions of the present invention contain water. Microemulsions of the present invention are transparent or translucent, optically isotropic and thermodynamically stable mixtures of oil and water
20 stabilized by surfactants and perhaps co-surfactants. The particle size of the dispersed phase of a microemulsion is about 100 to about 2000 angstroms, more preferably are about 100 to about 1000 angstroms. They can form spontaneously or with a little energy. Therefore they are simple to prepare
25 and are not process dependent i.e. the order of addition of starting materials or speed / type of mixing is not critical to the preparation of the microemulsions. It would be desirable to formulate antiperspirant compositions using microemulsions because microemulsions are easy and
30 inexpensive to process and can be inherently clear without

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requiring refractive index matching of the aqueous and non-aqueous phases.

Microemulsions have attracted considerable
5 technological and scientific interest. Water-in-oil (w/o) microemulsions containing water, an ionic surfactant, a cosurfactant and oil are the most investigated. The ionic surfactant- containing microemulsions usually exhibit stability over a large range of temperature. . When
10 inorganic salts are added, the minimum surfactant level to form water-in-oil microemulsions will increase. As the hydrocarbon oil chain length increases, the solubilization of aqueous phase into the oil phase decreases, while the liquid crystal area increases. Nonionic surfactant-
15 containing water-in-oil microemulsions require a large amount of surfactant as well. Unfortunately, nonionic surfactant-containing microemulsions commonly exhibit a small temperature range of stability

20 Microemulsions exist in the following forms: as water-in-oil, oil-in-water or as a *bicontinuous phase*, which is also called the *surfactant phase*. As used herein, the term "microemulsion means water-in-oil, oil-in-water or a bicontinuous phase, or mixtures thereof. Bicontinuous phase
25 microemulsions are found to solubilize a high amount of water and oil with lower levels of surfactant. The region around a bicontinuous phase microemulsion may transition into a swollen lamellar phase, otherwise known as a liquid crystal phase, and in certain cases these phases
30 (microemulsion and liquid crystal) may co-exist. These phases exhibit birefringence, shear induced (streaming)

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birefringence, and are thixotropic, viscoelastic and transparent. Because some of these systems exhibit increased viscosity the technical literature may refer to them as microemulsion gels.

5

It is an object of the present invention to provide antiperspirant compositions, which contain high levels of antiperspirant salts, cosmetic oils and surfactants suitable for application to the axilla. It is also an object of the present invention to provide antiperspirant compositions that do not require refractive index matching of the aqueous and nonaqueous phases in order to be clear. It is also an object of the present invention to provide microemulsion antiperspirant compositions that require little energy to manufacture. These and other objects of the present invention will become more readily apparent in the present application.

Patents and patent documents, which are cited in connection with the disclosed invention, are as follows:

DE 196 42 090 A1 discloses cosmetic or dermatologic compositions based on microemulsions.

25 U.S. Patent 5,162,378 discloses water in oil microemulsions comprising cetyl dimethicone copolyol, water, silicone, alcohol, and 5-40% by weight of one or more salts.

U.S. Patent 5,705,562 discloses a method of spontaneously forming a highly stable clear microemulsion by combining water, a volatile cyclic methyl siloxane or a

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volatile linear methyl siloxane and a silicone polyether surfactant. U.S Patent 5,707,613 is in the same patent family as the just mentioned patent.

5 WO 94/22420 is concerned with silicone-based skin care products, which are applied to the skin as aerosols and form a clear gel on the skin.

10 WO 94/19000 discloses pharmaceutical compositions in the form of a microemulsion which comprise an oil, a mixture of high and low HLB surfactants in which the high HLB surfactant comprises an aliphatic, aryl or aliphatic-aryl sulfate or sulfosuccinate or salt thereof, an aqueous phase and a biologically active agent.

15

WO 94/08610 discloses pharmaceutical compositions in the form of microemulsions which comprise an oil, a mixture of high and low HLB surfactants in which the high HLB surfactant comprises a medium-chain fatty acid salt, an aqueous phase and a biologically active agent.

20

U.S. 5,575,990 discloses roll-on antiperspirant compositions which are clear and, when applied to the human skin, do not leave a visible white residue after drying.

25 The clear antiperspirant roll-on compositions are stable under varying temperature conditions and provide a suitable cosmetically acceptable feel or sensation when applied to the human skin.

30 U.S. 5,487,887 discloses roll-on antiperspirant compositions and more particularly concerns antiperspirant

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compositions which are clear and stable under varying temperature conditions and, when applied to the human skin, do not leave a visible white residue after drying. The compositions in the form of an oil-in-water microemulsion, 5 comprise an antiperspirant active 5-30, PEG-7-glyceryl cocoate 5-25, emollients 0.5-3, cyclomethicone 3-7, and water 53-60%.

10 Summary of the Invention

The invention relates to a composition in the form of a microemulsion comprising an antiperspirant salt, a cosmetic oil, and a combination of at least one cationic quaternary 15 surfactant and at least one nonionic surfactant.

Detailed Description of the Invention

20 The present invention is directed to antiperspirant salt-containing microemulsions that are stable and clear liquids, or clear antiperspirant gels.

Stable clear microemulsions containing cosmetic oils, 25 antiperspirant salt, water, quaternary surfactants and nonionic surfactants have been discovered. The microemulsions are primarily composed of bicontinuous phase but the compositions include water-in-oil, oil-in-water, and microemulsion gels (liquid crystals). The microemulsions 30 are novel antiperspirant compositions that can be used in different types of applicators such as roll-on, sponge,

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mousse, pad, wipe, brush, gel and aerosol or non-aerosol spray applicators.

The microemulsions discovered in this invention contain
5 inorganic salts such as antiperspirant salts and cosmetic oils and the solubilization of high levels of both oil and aqueous solution of salts is achieved by incorporating combinations of a quaternary ammonium surfactant and a nonionic surfactant.

10

More specifically, the invention relates to a composition in the form of a microemulsion comprising an antiperspirant salt, cosmetic oils, and a combination of at least one cationic quaternary surfactant and at least one
15 nonionic surfactant.

The invention also relates to a method for controlling or preventing underarm perspiration and malodor, which comprises applying to the underarm area a composition
20 according to the invention.

The characteristics of the microemulsions of this invention include one or more of:

- 25
- The microemulsions exhibit stability over a relatively large range of temperature.
 - The viscosity ranges from a thick gel to a low viscosity sprayable liquid.
 - The types of the microemulsions formed are dependent on
30 the ratio of aqueous phase to the nonionic surfactant(s) and oil. When the percentage of the salt solution

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containing quaternary surfactant increases, the microemulsion changes from water-in-oil to oil-in-water type, and a bicontinuous microemulsion phase, or possibly a liquid crystal phase, will form in-between.

- 5 • The microemulsions can contain a high level of inorganic salts.
- The microemulsions contain a quaternary surfactant and a nonionic surfactant.
- The microemulsions contain cosmetically acceptable oils.
- 10 • A method for controlling or preventing underarm perspiration and malodor, which can be applied to the underarm area.
- The application of the microemulsions can be accomplished by using various product dispensers.

15

As used herein % means weight percent unless otherwise specified.

As used herein the term cationic surfactant means
20 quaternary ammonium surfactant.

The starting materials set forth herein are either known or can be prepared according to known methods. The compositions of the invention can be made by known methods
25 or by methods that are analogous to known methods.

As used herein, microemulsions mean stable clear microemulsions containing cosmetic oil; antiperspirant salts, water and surfactants. The microemulsions described
30 herein are primarily composed of bicontinuous phase but the

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compositions can include water-in-oil microemulsions. The compositions of the invention can also comprise a liquid crystal (that is, a microemulsion gel). More specifically, the compositions of the invention are selected from the group consisting of a microemulsion, a liquid crystal (that is, microemulsion gel), or a mixture of a microemulsion and a liquid crystal. The compositions of the invention comprise an antiperspirant salt, a cosmetic oil, and a combination of at least one cationic quaternary surfactant and at least one nonionic surfactant.

The compositions of the invention are novel antiperspirant compositions that can be used in different types of applicators such as roll-on, sponge, mousse, pad, brush, wipe, gel and aerosol or non-aerosol spray applicators.

All of the microemulsion compositions described contain antiperspirant salts and are clear and stable over a larger temperature range from room temperature to 45°C-50°C. The viscosity of some of the water-in-oil microemulsions are less than 10cst, therefore they are spray-able.

The invention relates to a composition in the form of a microemulsion comprising an antiperspirant salt, cosmetic oils, and a combination of at least one cationic quaternary surfactant and at least one nonionic surfactant.

A description of the ingredients included in the compositions of the invention now follows.

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Antiperspirant Salts

Antiperspirant salts contained in these microemulsions include, but are not limited to, aluminum chlorohydrate, aluminum dichlorohydrate, aluminum sesquichlorohydrate, aluminum chlorohydrate propylene glycol complex, aluminum dichlorohydrate propylene glycol complex, aluminum sesquichlorohydrate propylene glycol complex, aluminum chlorohydrate polyethylene glycol complex, aluminum dichlorohydrate polyethylene glycol complex, aluminum sesquichlorohydrate polyethylene glycol complex, aluminum zirconium trichlorohydrate, aluminum zirconium tetrachlorohydrate, aluminum zirconium pentachlorohydrate, aluminum zirconium octachlorohydrate, aluminum zirconium trichlorohydrate glycine complex, aluminum zirconium tetrachlorohydrate glycine complex, aluminum zirconium pentachlorohydrate glycine complex, aluminum zirconium octachlorohydrate glycine complex, aluminum chloride or buffered aluminum sulfate.

20

Antiperspirant actives for use herein are often selected from astringent active salts, including in particular aluminum, zirconium and mixed aluminum/zirconium salts, including both inorganic salts, salts with organic anions and complexes. Preferred astringent salts include aluminum, zirconium and aluminum/zirconium halides and halohydrate salts, such as chlorohydrates.

Aluminum halohydrates are usually defined by the general formula $Al_2(OH)_xQ_y$ or a hydrate thereof in which Q represents chlorine, bromine or iodine, x is variable from 2

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to 5 and $x+y=6$. The level of hydration is variable for example wherein there are up to about 6 or higher water molecules.

5 Zirconium actives can usually be represented by the empirical general formula: $ZrO(OH)_{2n-nz}B_z$ or a hydrate thereof in which z is a variable in the range of from 0.9 to 2.0 so that the value $2n-nz$ is zero or positive, n is the valence of B , and B is selected from the group consisting of
10 chloride, other halide, sulphamate, sulfate and mixtures thereof. Possible hydration to a variable extent is represented by wH_2O . It is preferable that B represents chloride and the variable z lies in the range from 1.5 to 1.87. In practice, such zirconium salts are usually not
15 employed by themselves, but as a component of a combined aluminum and zirconium-based antiperspirant. The level of hydration is variable for example wherein there are up to about 6 or higher water molecules.

20 The above aluminum and zirconium salts may have coordinated and/or bound water in various quantities and/or may be present as polymeric species, mixtures or complexes. In particular, zirconium hydroxy salts often represent a range of salts having various amounts of the hydroxy group.
25 Zirconium aluminum chlorohydrate may be particularly preferred.

Antiperspirant complexes based on the above-mentioned astringent aluminum and/or zirconium salts can be employed.
30 The complex often employs a compound with a carboxylate group, and advantageously this is an amino acid. Examples

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of suitable amino acids include dl-tryptophan, dl- β -phenylalanine, dl-valine, dl-methionine and β -alanine, and preferably glycine, which has the formula $\text{CH}_2 (\text{NH}_2) \text{COOH}$.

5 Complexes of a combination of aluminum halohydrates and zirconium chlorohydrates with or without with amino acids such as glycine can be employed in this invention. Certain of those Al/Zr-glycine complexes are commonly called ZAG in the literature. Aluminum-Zirconium actives or ZAG actives
10 generally contain aluminum, zirconium and chloride with an Al/Zr ratio in a range from 2 to 10, especially 2 to 6, an Al/Cl ratio from 2.1 to 0.9. ZAG actives also contain a variable amount of glycine. In certain conditions, salts with an Al/Zr ratio greater than 2 (also known as low-
15 zirconium actives) may be preferred. Actives of these preferred types are available from Westwood, from Summit and from Reheis.

Other antiperspirant-salt actives that may be utilized
20 include astringent titanium salts, for example those describe in GB 2299506A.

The proportion of solid antiperspirant salt in a composition normally includes the weight of any water of
25 hydration and any complexing agent that may also be present in the solid active. However, when the salt is in solution, its weight excludes any water present.

The antiperspirant active will often provide from 1 to
30 60% by weight of the aqueous phase, particularly from 10% to 60% of the aqueous phase. The final content of the salts in

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the formulations can range from 0.1% to 40% but 5-35% is preferred.

Other Aqueous Phase Ingredients

5

In addition to aluminum salts, the microemulsions, discovered in this invention, could solubilize aqueous solutions of monovalent, divalent and trivalent salts. The salts include sodium chloride, sodium sulfate, calcium chloride, calcium sulfate, magnesium chloride, aluminum sodium lactate, and mixtures thereof.

Other ingredients which can be dissolved in the aqueous phase include buffers, glycols, sugars, cyclodextrins, preservatives, antimicrobials, fragrances, chelating agents, amino acids, antimicrobials, anticholinergics, water-soluble polymers etc.

Water Content

20

The antiperspirant salts or other aqueous phase ingredients can be dissolved into water first and then combined with the non-aqueous phase. Water content in the final formulations can range from 1% to 60%, 5% to 30% is preferred and 10% to 25% is the most preferred.

Oil Phase

The oil phase of the compositions of the invention can contain cosmetic oils such as esters, ethers, long chain alcohols or ethoxylated alcohols, hydrocarbons, fatty acids,

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monoglycerides, diglycerides or triglycerides, fragrances, volatile or non-volatile silicone fluids. Cholesterol and some other lipids can be incorporated with the oil phase to act as emollients. The oil phase concentration can range
5 from 0% to 95%, but 20% to 60% is preferred.

Silicone fluids that may be included in compositions of the invention include volatile and non-volatile silicone fluids such as cyclomethicones and dimethicones.

10

Non-volatile silicones such as phenyl tris(trimethylsiloxy)silane can be included in compositions of the invention.

15 Silicone elastomers such as DC 9040, or DC 9010 by Dow Corning or GE SFE 839 by General Electric, can be included in the compositions of the invention.

20 Esters selected from the group consisting of cetyl octanoate, C12 -15 alcohol benzoate, isostearyl benzoate, diisopropyl adipate, isopropyl palmitate, isopropyl myristate and mixtures thereof may be included in the compositions of the invention.

25 Hydrocarbon oils such as aliphatic hydrocarbons (Permethyl 102A TM, Permethyl 101TM); hydrogenated polybutenes; hydrogenated polydecenes (SilkfloTM); dioctylcyclohexane; mineral oil, cyclohexane and mixtures thereof may be included in the compositions of the
30 invention.

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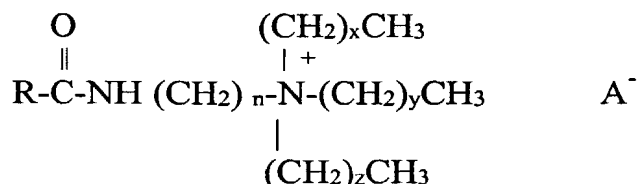
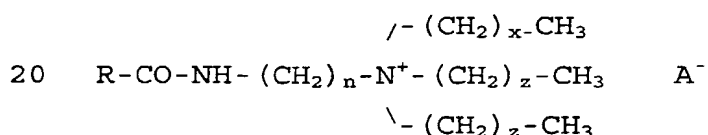
SurfactantsQuaternary Ammonium Surfactants

5 Combinations of a cationic, quaternary ammonium surfactant(s) and a nonionic surfactant are employed in the compositions of the invention.

10 The quaternary surfactant in this invention is essential, without which the formulation will be either extremely sensitive to temperature or a microemulsion will not form. The preferred cationic surfactants employed in compositions of the invention are alkylamidopropyl alkyldimonium quaternaries.

15

The preferred cationic quaternary surfactants have the following structure:-



wherein n is one to six.

x is zero to three

25 y is zero to three

z is zero to three

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with the proviso that $x+y+z \leq 6$

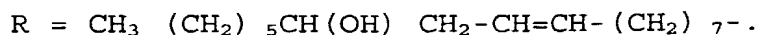
A⁻ is any physiologically acceptable counter ion which does not adversely affect the composition, and more specifically A⁻ can be selected from the group consisting of chloride, 5 bromide, ethosulfate, methyl sulfate, lactate, acetate, nitrate or sulfate.

where R is a ricinoleic derivative:

10 $\text{CH}_3 (\text{CH}_2)_5 \text{CH}(\text{OH}) \text{CH}_2 - \text{CH} = \text{CH} - (\text{CH}_2)_7 -$;
or mixtures thereof.

Obviously, variations on this structure, known to the art, can also be incorporated into embodiments of this 15 invention. The variations on surfactant structure should exhibit solubility in the aqueous antiperspirant salt solution. If the above mentioned solubility is maintained then variations in the quaternary ammonium salts can include but are not limited to, increasing or decreasing the alkyl 20 chain length, changing the position or removal of the hydroxyl group, changing the position or removing completely the double bond or combinations thereof.

The most preferred quaternary surfactant is 25 ricinoleamidopropyl ethyldimonium ethosulfate a compound according to the formula above wherein $n=3$, $x=1$, $y=0$, $z=0$, A⁻ = ethosulfate and



30 The surfactant described just above is also known, under the following trade names, as Surfactol Q4 from

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CasChem Inc., Lipoquat R from Lipo Chemicals or Mackernium DC-159 from McIntyre Chemical. Preferably the quaternary surfactant is supplied in a concentrated form (>90% active) with a low free amine content. This form is readily
5 miscible with the aqueous antiperspirant-salt solution.

The quaternary surfactant(s) in the compositions of the invention range from 0.1% to 30%, where 2% to 15% is preferred.

10

Nonionic Surfactants

The nonionic surfactant or co-surfactants employed in the compositions of the invention can be polyethoxylated
15 alcohol ethers or esters, polyglycerol mono or di-esters, glyceryl esters or branched guerbet ethoxylates or alcohols, or long chain carboxylic acids or combinations thereof. These compounds have a hydrophilic-lipophilic balance of between about 2 to about 15 and preferably less than about
20 12. Non-limiting examples are polyglycerol-3 diisostearate; glycerol oleate; poly glycerol-2 monoisostearate; polyglycerol -2 diisostearate, glyceryl isostearate. The most preferred ones are polyglyceryl-3 diisostearate, glyceryl isostearate and glycerol oleate or combinations
25 thereof.

The ratio of cationic surfactant to aqueous phase containing antiperspirant salt ranges from 30/70 to 4/96, the ratio from 10/90 to 5/95 is preferred. The ratio of
30 aqueous phase including salts, water and cationic surfactant

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to nonionic surfactant is 90/10 to 70/30, and the ratio from 90/10 to 80/20 is preferred.

Formulation Examples

5

The following samples are stable for one month at room temperature. The particle size or domain length of these compositions are between about 150 to about 600 angstroms. All samples are clear. Some samples exhibit streaming
10 birefringence. Some samples exhibit birefringence. The viscosity of these samples range from a thin liquid to a gel. These microemulsions are primarily composed of bicontinuous phase but the compositions include water-in-oil, and microemulsion gels (liquid crystals).

15

The following formulation examples are illustrative of the invention.

The following is a general formula for an
20 antiperspirant microemulsion of the present invention.

25

30

General Formulation Example:

Components		Specific Examples of components	Range	Preferred range
Oil Phase*		Aliphatic Hydrocarbon 90-10% Volatile Silicone 10-90%	0-95%	20-60%
Aqueous Phase*	Water	Deionized Water	1-60%	5-30%
	Antiperspirant-Salt	ACH or AZG or other salts	0.1-40%	5-35%
Non-ionic surfactant		Polyglycerol-3 diisosterate	0.2 to 30%	4-15% 5-10% most preferred
Cationic Quaternary Ammonium Surfactant		Ricinoleamidopropyl ethyl dimonium ethosulfate	0.1-30%	2-15%

- 5 *Cosmetic additives or other optional ingredients can be added to either phase as required.

Generalized manufacturing procedure:

- 10 1. Weigh all the oil phase components into a suitable vessel and mix until homogenous. Heat may be used to expedite dispersion of components solid at room temperature.

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2. The aqueous phase is prepared by mixing the quaternary ammonium surfactant with the antiperspirant salt solution.
3. Add the oil and water phases together and mix until a clear, homogenous dispersion is formed.
- 5 4. The microemulsion formulation is transferred into a suitable dispenser or applicator.

The following examples more fully illustrate embodiments of this invention, all percentages being by weight unless
10 otherwise noted. The following specific examples, which are compositions of the invention, were made.

Compositions were prepared according to the following procedure:

15

1. Mix the cationic surfactant with the antiperspirant salt solution
 2. Mix the nonionic surfactant with the oil mixture, then add the two mixtures together and mix well.
 - 20 3. Heat may be applied to better dissolve solid nonionic surfactants, which are solid such as glyceryl oleate, in the oil phase prior to mixing the aqueous and non-aqueous phases
-

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4.

	Prisorine 3700 %	Cationic ** %	Aluminum Zirconium tetra %	Water%	DC245%	HC* %	
1	10.03	5.98	13.55	20.31	15.04	35.09	
2	8.99	4.66	10.57	15.85	17.98	41.95	
3	7.02	3.45	7.82	11.74	20.99	48.98	
4	3.97	1.73	3.93	5.91	25.34	59.12	
5	Prisorine 3700 %	Cationic ** %	ACH %	Water %	DC245%	HC * %	
6	9.97	6.78	19.2	19.2	13.45	31.40	
7	2.99	1.02	2.89	2.90	27.06	63.14	
	Glyceryl oleate %	Cationic ** %	Aluminum Zirconium tetra %	Water %	DC 245 %	HC * %	
8	14.24	11.71	22.09	33.13	5.65	13.18	
9	11.05	8.55	16.13	24.20	12.02	28.05	
10	10.02	7.89	14.88	22.33	13.46	31.42	
11	9.99	6.98	13.17	19.75	15.03	35.08	Birefringent
12	14.95	12.27	23.13	34.69	4.49	10.47	
	Glyceryl oleate %	Cationic ** %	ACH %	Water %	DC 245 %	HC * %	
13	3.99	12.91	36.57	36.57	2.99	6.97	
14	2.99	1.83	5.17	5.18	25.45	59.38	Birefringent
15	8.50	7.70	21.82	21.82	12.05	28.11	
	Prisorine 3700 %	Cationic ** %	Aluminum Zirconium penta %	Water %	DC245%	HC * %	
16	16.64	8.67	23.2	34.8	5.01	11.68	Birefringent
17	14.12	6.04	16.17	24.25	11.83	27.59	Birefringent
18	7.46	4.87	16.30	16.29	16.52	38.56	

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	Glyceryl isostearate %	Cationic ** %	Alumin um Zircon ium penta %	Water %	DC 245 %	HC * %	
19	11.02	11.09	25.15	37.72	4.51	10.51	Birefri ngent
20	10.02	8.99	20.37	30.55	9.02	21.05	Birefri ngent
21	9.03	7.64	17.32	25.99	12.00	28.02	Birefri ngent
22	7.97	6.32	14.32	21.47	14.98	34.94	
23	6.02	3.60	8.15	12.22	21.00	49.01	
	Glyceryl isostearate %	Cationic ** %	Alumin um Zircon ium penta %	Water %	DC 245 %	HC * %	
24	6.02	4.434	7.82	11.72	21.00	49.01	
25	8.52	13.64	24.03	36.05	5.33	12.43	
26	9.00	5.71	8.72	16.46	18.03	42.08	
26	4.68	0.14	0.25	0.38	28.36	66.19	
27	9.74	0.46	0.81	1.21	26.33	61.45	
	Glyceryl isostearate %	Cationic ** %	Alumin um Zircon ium penta %	Water %	DC 245 %	HC * %	
28	11.47	11.80	26.76	40.13	2.95	6.89	Birefri ngent
29	11.11	11.07	25.10	37.65	4.52	10.55	
30	10.03	6.74	15.29	22.93	13.50	31.51	
31	9.54	6.06	13.73	20.60	15.02	35.05	
32	11.38	11.91	27.00	40.51	2.76	6.44	
	Glyceryl isostearate %	Cationic ** %	Alumin um Zircon ium penta %	Water %	DC 245 %	Silkofl o 366-NF %	
33	7.45	16.94	30.34	44.66	0.43	0.18	Birefri ngent
34	12.36	11.88	22.40	33.59	13.85	5.92	Birefri ngent
35	12.06	11.92	22.47	33.71	13.89	5.95	
36	12.05	9.26	17.46	26.19	24.53	10.51	
37	10.93	7.78	14.67	22.01	31.23	13.38	

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	Prisorine 3700 %	Cationic ** %	Aluminum Zirconium penta %	Water %	DC 245 %	Silkflo 366-NF %	
38	10.67	11.19	25.36	38.05	10.31	4.42	Birefringent
39	14.01	9.89	22.41	33.61	14.06	6.02	
40	4.93	2.22	5.03	7.55	56.20	24.07	
41	13.98	6.90	15.64	23.45	28.02	12.01	
42	11.51	5.77	13.08	19.62	35.02	15.00	
43	9.51	4.58	10.37	15.56	41.98	18.00	
44	7.98	3.32	7.52	11.28	48.93	20.97	
	Prisorine 3700 %	Cationic ** %	Aluminum Zirconium penta %	Water %	DC 245 %	Silkflo 366-NF %	
45	11.05	13.48	25.42	38.08	8.34	3.63	
46	12.03	11.91	22.46	33.70	13.92	5.98	
47	11.96	9.80	18.49	27.73	22.41	9.61	Birefringent
48	15.96	11.22	21.16	31.73	13.95	5.98	Birefringent
49	14.03	9.78	18.44	27.66	21.06	9.03	
	Isofol 12 alcohol ethoxylate/ cholesterol	Cationic ** %	ACH %	Water %	DC245%	HC * %	
50	20.15/0	8.21	23.26	23.26	7.52	17.60	Birefringent
51	12.71/2.44	6.72	18.85	18.85	12.12	28.31	

* HC means hydrocarbon: Permethyl 102A, listed in the above table

** Cationic means the cationic surfactant:

5 Ricinoleamidopropyl ethyldimonium ethosulphate

Further examples include:

Example 52

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Ingredient (INCI)	Trade Names	Source	Percent
Ricinoleamidopropyl Dimonium Ethosulfate	Surfactol Q4	CasChem, Inc	7.50%
Polyglycerol-3 Diisostearate	Prisorine PG3 DI 3700	Uniqema	10%
Aliphatic Hydrocarbon	Permethyl 102A	Presperse	28%
Cyclopentasiloxane	DC245	Dow Corning	12%
Aluminum Chlorohydrate 50%	Westchlor 200	Westwood	42.50%
		Total:	100%

Example 53

Ingredient (INCI)	Trade Names	Source	Percent
Ricinoleamidopropyl Dimonium Ethosulfate	Surfactol Q4	CasChem, Inc	7.50%
Glyceryl Isostearate	Peceol Isostearique	Gattefoss e	10%
Hydrogenated Polydecene	Silkflo 366	Lipo Chemicals	12%
Cyclopentasiloxane	DC245	Dow Corning	28%
Aluminum Zirconium Pentachlorohydrate 40%	Low Zirconium Penta Solution R280-130	Reheis	42.50%
		Total:	100%

Example 54

Ingredient (INCI)	Trade Names	Source	Percent
Ricinoleamidopropyl Dimonium Ethosulfate	Surfactol Q4	CasChem, Inc	2.77%
Aluminum Zirconium Pentachlorohydrate 40%	Low Zirconium Penta Solution R280-130	Reheis	47.63%
Glyceryl Isostearate	Peceol Isostearique	Gattefoss e	3.06%
Hydrogenated Polydecene	Silkflo 366	Lipo Chemicals	11.70%
Cyclopentasiloxane	DC245	Dow Corning	27.04%
Ethoxylated Guerbet Alcohol C14 / 4 EO HLB ~9	Novel II Isofol 14T+4EO	Condea Vista	7.80%
		Total:	100%

5 Examples 55 and 56

Ingredient (INCI)	Trade Names	Supplier	55	56
			Per-cent	Per-cent
Ricinoleamidopropyl ethyl dimonium ethosulfate	Surfactol Q4	Caschem	2.32	2.83
Aluminum zirconium penta chlorohydrate	Rezal 67	Reheis	15.94	18.13
Water	Deionized Water	Stock	23.91	27.19
Urea	Urea	Janssen Chimica	-	3.34
Cyclopentasiloxane	DC 245	Dow Corning	29.08	22.08
Polydecene hydrogenated	Silkflo366NF	Lipo Chemicals	11.62	9.46
Glyceryl isostearate	Peceol isostearique	Gattefosse	5.26	-
Polyglyceryl-3 diisostearate	Prisorine 3700	Unichema	0.87	3.49
Ethoxylated Guerbet alcohol C ₁₈ EO ₁₀	Novel II I18T-10 ethoxylate	Condea Vista	3.36	6.19
2-hexyldecanol (Guerbet C16 Alcohol)	Isoflo 16	Condea Vista	7.64	7.29
		Total	100	100

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Raw materials used in preparation of the example compositions of the invention are as follows:

Trade Name	Chemical Name	Vender
DC 245	Cyclomethicone D5	Dow Corning
DC 344	Cyclomethicone D4	Dow Corning
Silkflo 364 or 366	Hydrogenated Polydecene	Lipo Chemical
Permethyl 102 A	Aliphatic hydrocarbon	Permethyl Specialties
Permethyl 101	Aliphatic hydrocarbon	Permethyl Specialties
Trivent OC-16	Cetyl octanoate	Trivent Chemical Company
Cetiol S	Dioctyl cyclohexane	Henkel Corporation
Peceol Isostearique	Glyceryl isostearate	Gattefosse
Monomuls 90-018	Glycerol oleate	Henkel Corporation
Fancol Polyiso 275	Hydrogenated polyisobutene	The Fanning Corp.
Finsolve TN	C12-C15 alcohol benzoate	Finetex
Finsolve SB	Isostearyl benzoate	Finetex
Prisorine 3700	Polyglycerol -3 Diisostearate	Unichema North America
Prisorine 3792	Polyglycerol-2 diisostearate	Unichema North America
Prisorine 3791	Polyglycerol-2 monoisostearate	Unichema North America
Glucate DO	Methyl glucoside dioleate	Amercol
Glucate SS	Methyl glucoside sesquistearate	Amercol

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Estol 3609	Glycerol tri-2-ethylhexanoate	Unichema North America
Dow Corning 556	Phenyl tris(trimethylsiloxy)silane	Dow Corning

Trade Name	Chemical Name	Vender
Ceraphyl 230	Diisopropyl Adipate	ISP Van Dyk Inc
Mineral oil	Hydrocarbon	Witco
Novel II 12-5 Ethoxylate	Ethoxylated alcohol or Branched Guerbert ethoxylate	Condea Vista Company
Cholesterol	Cholesterol	Rita Corporation
Surfactol Q4	Ricinoleamidopropyl dimonium sulfate	CasChem
Westchlor 200 50% w/w	Aluminum chlorohydrate (ACH)	West Wood
Low zirconium penta solution R280-130 40%w/w	Low zirconium: Aluminum Zirconium Pentachlorohydrate	Reheis
Rezal 67 Solution 40%w/w	Aluminum Zirconium Pentachlorohydrate (penta)	Reheis
Westchlor Zr 44 50% w/w	Aluminum Zirconium tetrachlorohydrate (tetra)	West Wood
Westchlor Zr 41 45%w/w	Aluminum Zirconium tetrachlorohydrate-glycine	West Wood

5

The foregoing description and examples illustrate selected embodiments of the present invention. In light thereof, various modifications would be suggested to one skilled in the art, all of which are within the spirit and scope of this invention.

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Claims:

1. A composition which is selected from the group consisting of a microemulsion, a liquid crystal, or a mixture of a microemulsion and a liquid crystal which comprises an antiperspirant salt, a cosmetic oil, and a combination of at least one cationic quaternary surfactant and at least one nonionic surfactant.
2. A composition in accordance with claim 1 characterised in that said antiperspirant salt is selected from the group consisting of aluminum, zirconium and mixed aluminum/zirconium salts.
3. A composition in accordance with claim 1 or 2 characterised in that said antiperspirant salt is a zirconium salt complexed with aluminum salts having coordinated or bound water.
4. A composition in accordance with any preceding claim characterised in that said antiperspirant salt is present in the aqueous phase at from about 1 to about 60%.
5. A composition in accordance with claim 4 characterised in that said antiperspirant salt is present in the aqueous phase at from 10% to about 60%.
6. A composition in accordance with any preceding claim characterised in that said aqueous phase further comprises a buffer, a glycol, a sugar, a cyclodextrin,

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a preservative, an antimicrobial, a chelating agent, a water-soluble polymer, an anticholinergic, a monovalent salt, a divalent salt, a trivalent salt, fragrances or mixtures thereof.

5

7. A composition in accordance with any preceding claim in which said aqueous phase is present at about 1% to about 60%, more preferably at 5% to 30%, and most preferably at 10 to 25%.

10

8. A composition in accordance with any preceding claim characterised in that said cosmetic oil comprises esters, ethers, long chain alcohols or ethoxylated alcohols, hydrocarbons, fatty acids, monoglycerides, diglycerides triglycerides, fragrances and volatile or non-volatile silicone fluids, and cholesterol.

15

9. A composition in accordance with claim 8 characterised in that said oil phase comprises silicone fluids which in turn comprise a volatile or non-volatile silicone such as cyclomethicone or dimethicone.

20

10. A composition in accordance with claim 8 or 9 characterised in that said non-volatile silicone is phenyl tris(trimethylsiloxy)silane.

25

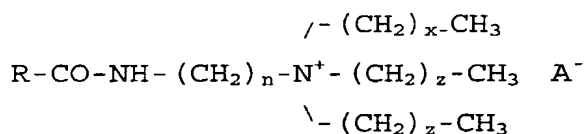
11. A composition in accordance with claim 8 characterised in that said esters are selected from the group consisting of cetyl octanoate, C12 -15 alcohol benzoate, isostearyl benzoate, diisopropyl adipate and mixtures thereof.

30

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12. A composition in accordance with claim 8 wherein said hydrocarbon fluids are selected from the group such as aliphatic hydrocarbons; hydrogenated polydecenes; hydrogenated polybutenes; dioctylcyclohexane; mineral oil, cyclohexane and mixtures thereof.

13. A composition in accordance with any preceding claim characterised in that the cationic quaternary ammonium surfactant has the following structure:



wherein n is one to six.

x is zero to three

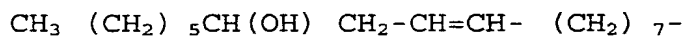
y is zero to three

z is zero to three

with the proviso that $x+y+z \leq 6$

A^- is any physiologically acceptable counter ion which does not adversely affect the composition, and more specifically A^- can be selected from the group consisting of chloride, bromide, ethosulfate, methyl sulfate, lactate, acetate, nitrate or sulfate.

where R is a ricinoleic derivative:



Or mixtures thereof.

14. A composition in accordance with claim 13 wherein $n=3$, $x=1$, $y=0$, $z=0$, A^- = ethosulfate and $R = CH_3-(CH_2)_5-CH(OH)-CH_2-CH=CH-(CH_2)_7-$.

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15. A composition in accordance with any preceding claim characterised in that said cationic quaternary ammonium surfactant is present at 0.1% to 30%, more preferably at 1% to 30%, most preferably at 2% to 15%.

5

16. A method for controlling or preventing underarm perspiration and malodor which comprises applying, to an underarm, an effective amount of a composition of claim 1.

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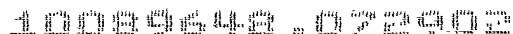
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(54) Title: ANTIPERSPIRANT COMPOSITIONS COMPRISING MICROEMULSIONS

(57) Abstract: Stable, clear, antiperspirant microemulsions containing cosmetic oils, antiperspirant salts, and water and combinations of cationic quaternary ammonium salt are provided. These microemulsions can be used in different types of applicators such as roll-on, sponge, mousse, pad, brush, gel and aerosol or non-aerosol spray applicators.

WO 01/24766 A1



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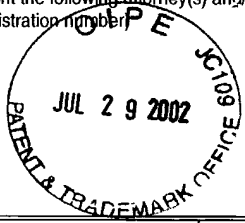
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SIGNATURE OF INVENTOR 201 	SIGNATURE OF INVENTOR 202 	SIGNATURE OF INVENTOR 203
DATE 5/15/02	DATE May 15, 2002	DATE